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⑯ Method of lubricating an elastic fibrous material.

⑯ A lubricant which comprises a polyether polyol which is a reaction product of  
i) one or more C<sub>2</sub>-4-alkylene oxides and  
ii) a polyhydroxy compound selected from pentites, hexites, monosaccharides or disaccharides is used for lubricating an elastic fibrous material.

EP 0 611 049 A1

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The present invention relates to a method of lubricating an elastic fibrous material and to an elastic fibrous material incorporating a lubricant.

In the commercial production of yarns, such as continuous-filament yarns, it is common practice to apply lubricants to filaments to reduce the tendency towards breakage of the individual filaments when they are subjected to various mechanical strains. Excessive broken filaments result in a loss of yarn strength or even cause interruption of the manufacturing process. The lubricants are commonly applied during the spinning, twining, knitting or weaving process. Due to the ever-increasing processing speed sought by textile fiber manufacturers and processors, very efficient lubrication of the fibers is required to minimize fiber or yarn breakage. Various compositions have been suggested as textile lubricants.

U.S. patent 3,341,452 discloses a high temperature lubricant composition for textile fibers comprising 40 to 60 weight parts of coconut oil, 5 to 20 weight parts of a soft hydrocarbon-wax and 25 to 50 weight parts of a nonionic emulsifier. The soft wax is of mineral oil origin. Disclosed as a non-ionic emulsifier is a condensate of 10 mols of ethylene oxide and 1 mol of nonylphenol and a mixed oleic/lauric acid ester of the condensate of 30 mols of ethylene oxide and 1 mol sorbitol. The lubricant composition is dispersed in water to form an aqueous emulsion which is then applied to textile yarns. It is said that the treated yarn has outstanding high-temperature processing properties.

After the yarn has been processed in various steps to an article, such as cloth etc., the lubricant is usually removed from the article. Coconut oil and hydrocarbon-waxes are water-insoluble and can only be removed from the article with considerable effort. Furthermore, mineral oil is hardly biodegradable and contaminates water which is usually used for washing the article.

U.S. patent 3,38,830 discloses a textile lubricant containing, on a weight basis, 50 to 80 parts of a polyoxyalkylene diol, a polyoxyalkylene monoether or a polyoxyalkylene monoester which contains oxyethylene groups and oxy-1,2-propylene groups in a ratio between 3:1 and 1:1 and has a molecular weight of at least about 5,000, 10 to 40 parts of an ester having a smoke point above 150°C and a viscosity at 25 °C between 25 and 400 poises, and 10 to 40 parts of an emulsifier. The U.S. patent discloses that the polyoxyalkylene preferably is a diol containing oxyethylene and oxy-1,2-propylene groups in a weight ratio of 3:1 and having a molecular weight of about 15,000. As examples of suitable esters are disclosed coconut oil, sperm oil, neopentyl glycol distearate, glycerol trioleate, trimethylol propane tristearate or pentaerythritol tetraoleate. Suitable emulsifiers are anionic and/or well known non-ionic emulsifiers, such as ethylene oxide condensates of phenol, aliphatic alcohols or acids having 12 to 20 carbon atoms, ethylene oxide condensates of polyols or their partial higher fatty acid esters, or partial higher fatty acid esters of polyol-ethylene oxide condensates. As preferred non-ionic emulsifiers are mentioned a condensate of 10 mols of ethylene oxide and 1 mol of nonylphenol and a mixed oleic/lauric acid ester of the condensate of 30 mols of ethylene oxide and 1 mol sorbitol. Unfortunately, it has been found that certain ethoxylated and/or propoxylated monoalcohols and diols decrease the elasticity of elastic fibers or yarns, such as polyurethane elastomer fibers or yarns. Apparently, these ethoxylated and/or propoxylated monoalcohols and diols swell or otherwise attack these elastic fibers.

U.S. patent No. 4,496,632 relates to a method of lubricating thermoplastic synthetic fibers, such as nylon, polyester, polyolefin, poly(benzimidazole), carbon or glass fibers with a modified polyoxyalkylene polyol. The U.S. patent discloses that a typical apparatus for spinning synthetic fibers includes many parts of polyurethanes elastomer. It is disclosed that the use of polyoxyalkylene compounds, such as block or heteric polymers of ethylene oxide and propylene oxide, as spin finishes results in swelling or softening of the polyurethane elastomers used in the spinning machine. In order to reduce this problem, the U.S. patent suggests modification of the polyoxyalkylene polyol by capping all of the hydroxyl groups with benzyl, aryl, substituted benzyl, or substituted aryl groups or alkyl groups having 1 to 4 carbon atoms.

U.S. patent No. 3,704,225 discloses a water emulsifiable textile finish composition for polyester yarns undergoing false twist texturing. The composition consists of 22 to 70 weight percent of a lubricant, 5 to 35 weight percent an antistatic agent, and 15 to 55 weight percent of an emulsifying agent. The lubricant is mineral oil, vegetable oil, a stearate ester or a pelargonate ester. The antistatic agent is preferably potassium hexyl phosphate. The emulsifying agent is polyoxyethylene(2)oleyl alcohol, sorbitan trioleate, polyoxyethylene(20)sorbitan trioleate, and partial oleic acid esters of ethylene oxide adducts of hexitols, such as sorbitol or hexitol-water mixtures. The textile finish composition is said not to attack the polyurethane belts and cots used in false twist texturing equipment.

U.S. patent 4,816,336 relates to a synthetic fiber containing from 0.08 to 0.15 weight percent of a primary fiber finish composition and from 0.01 to 0.06 weight percent of a certain secondary lubricant. The primary finish composition contains from 25 to 85 weight percent of at least one alkyl phosphate ester salt and from 15 to 75 weight percent of a quaternary trialkyl ethyl ammonium ethosulfate salt or a reaction product of a polyoxyethylene ether and a C<sub>6-22</sub> fatty acid. The U.S. patent suggests that the increased fiber-to-metal friction

may be reduced by the application of the secondary lubricant. However, the usage of a high level of alkyl phosphate esters salts may cause concern for environmental reasons.

U.S. patent No. 3,421,935 discloses a continuous filament, bulkable nylon yarn having on its surface a lubrication composition comprised of specific proportions of coconut oil, the tetraoleate-laurate pentaester of the condensate of one mol of sorbitol with about 30 mols of ethylene oxide, and the polyether condensate of nonylphenol and about 6 mols of ethylene oxide.

Presently lubricants based on mineral oil or silicone are commercially used for fibrous materials, such as polyurethane elastomer fibers, because mineral oil and silicone do not dissolve, swell or otherwise attack polyurethane elastomer fibers. However, mineral oil and silicone are hardly biodegradable, water-insoluble and can only be removed from articles made of the fibrous material with considerable effort. Yellowing of such articles, such as cloths, is often observed after removal of mineral oil or silicone.

One aspect of the present invention is a method of lubricating an elastic fibrous material which method comprises contacting a polymeric starting material for the fibrous material before or during the polymeric material is processed, preferably spun, to the elastic fibrous material

15 with a lubricant comprising a polyether polyol which is the reaction product of  
 i) one or more C<sub>2-4</sub>-alkylene oxides; and  
 ii) a polyhydroxy compound which is a pentite, a hexite, a monosaccharide or a disaccharide.

Another aspect of the present invention is a method of lubricating an elastic fibrous material which method comprises contacting the elastic fibrous material with the above-mentioned lubricant. The elastic fibrous material is preferably contacted with this lubricant before or whilst the elastic fibrous material is subjected to a twining process and/or before or whilst the elastic fibrous material is subjected to a knitting or weaving process.

Yet another aspect of the present invention is an elastic fibrous material incorporating a lubricant comprising a polyether polyol which is the reaction product of

25 i) one or more C<sub>2-4</sub>-alkylene oxides; and  
 ii) a polyhydroxy compound which is a pentite, a hexite, a monosaccharide or a disaccharide.

The lubricant for elastic fibrous material contains or consists of a polyether polyol. The polyether polyol is a reaction product of i) one or more C<sub>2-4</sub>-alkylene oxides and ii) at least one polyhydroxy compound which is a pentite, hexite, monosaccharide or disaccharide.

30 Reaction products of i) one or more C<sub>2-4</sub>-alkylene oxides; and ii) a polyhydroxy compound are generally known in the art.

French patent FR-A-1,501,916 discloses a process for producing polyether polyols by reacting an aldose or ketose containing up to 12 carbon atoms with at least one alkylene oxide, such as ethylene oxide or propylene oxide. 150-200% of the stoichiometric quantity of the polyol is used which is necessary to produce a monoether. It is disclosed to use the produced polyether polyols for the production of polyurethane foams.

35 German Offenlegungsschrift DE-A-1,285,663 discloses a process for producing regenerated cellulose yarns and fibers by spinning viscose in aqueous sulfuric acid, sodium sulfate and zinc sulfate wherein the viscose and/or the spin bath contain a modifying agent. The modifying agent is an ethoxylated carbohydrate, optionally mixed with a formaldehyde-releasing compound. The ethoxylated carbohydrate preferably is ethoxylated glucose, saccharose, sugar alcohols or beta- or gamma-cellulose. According the examples saccharose is utilized which has been ethoxylated with ethylene oxide at a molar ratio between ethylene oxide and saccharose of 20-40 : 1. The produced regenerated cellulose yarns and fibers are said to be of improved quality, particularly of improved strength and wet strength, of decreased swelling property and, accordingly, of improved form stability.

40 U.S. patent No. 3,370,056 discloses a process for producing polyoxyalkylene polymers wherein an alkylene oxide, such as ethylene oxide, propylene oxide, butylene oxide, etc., is reacted with an initiator having at least one active hydrogen atom in the presence of a solid catalyst. A large number of glycols, triols, tetrols, sugars, sugar alcohols, glycosides, primary and secondary amines and thioglycols are listed as an initiator. The produced polyoxyalkylene polymers are said to be useful as interface active agents or the raw materials therefor, textile auxiliary, lubricating oil, brake oil, oilment base mold release agent, and raw material for the manufacture of polyurethane resins. The produced tri- or more functional polyether polyols are said to be of high quality and especially desirable for manufacture of polyurethane foams.

45 However, none of the above-mentioned references suggests the use of a polyether polyol which is the reaction product of

50 i) one or more C<sub>2-4</sub>-alkylene oxides; and  
 ii) a polyhydroxy compound which is a pentite, a hexite, a monosaccharide or a disaccharide for lubricating an elastic fiber material.

55 Moreover, the applicant of the present invention has found that polyether polyols which have been pro-

duced by reacting a monofunctional alcohol or a diamine with an alkylene oxide, as suggested in U.S. patent 3,370,056, are nor useful for lubricating an elastic fiber material. Such polyether polyols cause loss of fiber elasticity, as will be shown in the Comparative Examples further below.

The applicant of the present invention has surprisingly found that

5 a polyether polyol which is the reaction product of i) one or more  $C_{2-4}$ -alkylene oxides; and ii) a polyhydroxy compound which is a pentite, a hexite, a monosaccharide or a disaccharide does not swell or otherwise notably attack an elastic fiber material, such as elastic polyurethane fibers. It has also been found that such a polyether polyol can be removed from articles which are at least partially made of an elastic fiber material without undue difficulties.

10 Preferred  $C_{2-4}$ -alkylene oxides for producing the polyether polyol which is used in the method of the present invention are 1,2- or 2,3-butylene oxide or, preferably, ethylene oxide or propylene oxide. The alkylene oxides may be used individually, alternatingly in sequence, or in mixtures for reacting with the polyhydroxy compound.

The polyhydroxy compound is a pentite, hexite, monosaccharide or disaccharide. Exemplary of useful pentites are lyxosite, xylite, arabinitite or ribite. Preferred hexites are sorbitol or mannitol. Preferred monosaccharides are ketopentoses, such as ribulose or xylulose; or aldopentoses, such as lyxose, xylose, arabinose or ribose; or aldohexoses, such as allose, altrose, glucose, mannose, galactose, idose, talose or galactose; or ketohexoses, such as psicose, sorbose, tagatose or fructose. The D-form of the monosaccharides are generally preferred. Preferred disaccharides are maltose, cellobiose, lactose or saccharose. The (+) forms of the disaccharides are generally preferred.

20 The molar ratio between the  $C_{2-4}$ -alkylene oxide(s) and the polyhydroxy compound preferably is from 4 : 1 to 30 : 1, more preferably from 6 : 1 to 18 : 1, most preferably from 8 : 1 to 15 : 1.

25 Processes for reacting one or more  $C_{2-4}$ -alkylene oxides and an above-described polyhydroxy compound as well as the produced polyether polyol are well known in the art. The weight average molecular weight of the polyether polyol preferably is from 300 to 2500, more preferably from 400 to 2000, most preferably from 500 to 1500. The preferred low average molecular weight is very advantageous because it improves the biodegradability of the polyether polyol.

30 The above-described polyether polyols have excellent lubricating properties. One or more of these polyether polyols may be used in their undiluted form for lubricating an elastic fibrous material. However, the lubricant which is used in the methods of the present invention preferably contains one or more of the described polyether polyols and a diluent. Preferably, the lubricant contains from 1 to 90 percent, more preferably from 4 to 70 percent, most preferably from 6 to 50 percent of the polyether polyol and preferably from 99 to 10 percent, more preferably from 96 to 30 percent, most preferably from 94 to 50 percent of one or more diluents, based on the total weight of polyether polyol and diluent. One useful diluent is water. The polyether polyol is generally miscible with water at any weight ratio. Other useful diluents are organic liquids, such as glycol diethers, preferably the mono-, di- or tripropylene glycol diethers, such as the  $C_{1-6}$ -alkyl diethers, preferably the dimethyl ethers, diethyl ethers, dipropyl ethers or dibutyl ethers thereof, such as the di-n-butyl ethers, or the mixed ethers thereof such as the methyl/n-butyl ethers of mono-, di- or tripropylene glycol. Other preferred organic liquids are glycol monoethers, such as mono-, di- or tripropylene glycol monoethers, such as the  $C_{1-6}$ -alkyl ethers, preferably the methyl, ethyl, n-propyl, i-propyl, n-butyl, sec.-butyl or tert.-butyl ethers of mono-, di- or tripropylene glycol, or the mono-, di- or triethylene glycol monoethers, such as the  $C_{1-6}$ -alkyl ethers, preferably the methyl, ethyl, n-propyl, i-propyl, n-butyl, sec.-butyl or tert.-butyl ethers of mono-, di- or triethylene glycol. Other useful diluents are glycols, such as mono-, di-, tri- or polypropylene glycol. Other useful diluents generally contain one or more hydroxyl, ester or carbonyl functionality. Exemplary thereof are white spirit, alcohols, such as saturated open-chain or cyclic alcohols, preferably methanol, ethanol, the propanols, such as n-propanol or isopropanol, the butanols, such as n-butanol or isobutanol, the hexanols, such as n-hexanol or cyclohexanol, the heptanols, octanols, decanols, dodecanols, such as lauryl alcohol, or the octadecanols, such as stearyl alcohol; or unsaturated alcohols, preferably the allyl or furfuryl alcohol, substituted or non-substituted phenols, such as phenol or the methyl phenols, or ketones, such as acetone, methyl ethyl ketone, methyl iso-butyl ketone or cyclohexanone, esters such as ethylene glycol ethoxy propionate or TEX-ANOL (trademark), which is a blend of various esters and which is commercially available from BASF. These diluents usually do not have lubricating properties but are useful for providing the desired viscosity to the lubricant composition.

45 A preferred lubricant composition contains

50 a) from 0 - 60 percent, preferably from 5 to 50 percent, more preferably from 15 to 40 percent of a glycol mono- or diether, an ester, a ketone or white spirit,  
b) from 1 to 80 percent, preferably from 10 to 70 percent, more preferably from 20 to 50 percent of a glycol, and

c) from 1 to 80 percent, preferably from 10 to 70 percent, more preferably from 20 to 50 percent of an above-described polyether polyol,  
 by the total weight of a), b), and c). Preferred glycol mono- or diethers, esters, ketones and glycols are described above. The preferred lubricant composition may contain one or more compounds selected from group a), one or more compounds selected from group b) and one or more compounds selected from group c). The viscosity of the lubricant composition preferably is from 5 to 75 cSt, more preferably from 10 to 50 cSt, most preferably from 15 to 35 cSt, measured at 40 °C.

The lubricant may contain optional additives, such as an anti-wear additive, an extreme-pressure additive, a textile softener, a wetting agent, an antistatic agent, an antioxidant, a bactericide and/or a fungicide and the like, provided that such additives do not influence the properties of the fibrous material to be lubricated in an undesirable manner. If such optional additives are contained in the lubricant, their amount generally is from 0.05 to 10 percent, preferably from 0.1 to 5 percent, based on the total weight of the lubricant.

The above described lubricant is very useful in the methods of lubricating an elastic fibrous material, particularly a synthetic elastic organic fibrous material. The term "elastic fibrous material" as used herein includes filaments, fibers, rovings, yarns, threads, strands and the like having an elongation at tear of at least 150%, preferably at least 200%, more preferably at least 400% and which have a substantially reversible extensibility. Elastic fibrous materials are generally designated as "elastomer fibers". The lubricant is useful for decreasing the fiber-to-fiber friction and the fiber-to-machine friction when the elastic fibrous material is produced from a polymeric material by a spinning operation or when the produced elastic fibrous material is further processed, for example in a twining process, in a step of producing yarns, threads or strands from fibers or filaments or in a knitting or weaving process. Furthermore, the described lubricant invention does not tend to dissolving, swelling or otherwise attacking an elastic fibrous material, such as a fibrous polyurethane elastomer material. Therefore, the elasticity of an elastic fibrous material, such as a fibrous polyurethane elastomer material, is not essentially decreased by applying the lubricant on to such fibrous material. The lubricant is preferably used for lubricating an elastic fibrous material which is partially or entirely made of a polyurethane elastomer. Fibrous polyurethane elastomer materials as well as their excellent elasticity are known in the art. Polyurethane elastomers can be used as such or in combination with one or more other synthetic organic materials, such as polyethylene, polypropylene, poly(vinyl chloride), such as Fibravyl or Leavil (trademarks), polyacrylonitrile, such as Dralon or Orion (trademarks), modacrylics (a copolymer of acrylonitrile and vinyl chloride), poly(vinyl alcohol), such as Kuralon (trademark), polyacryl, vinylate, trivinyl, 6-polyamide, such as Perlon (trademark), 6,6-polyamide, such as Nylon (trademark) or polyesters like poly(ethylene terephthalate), such as Dracon, Diolen, Terylene or Trevira (trademarks). Often a polyurethane elastomer content of 2 to 20 weight percent, preferably of 5 to 10 weight percent, is sufficient in textile fibers to make them highly elastic. Fibrous polyurethane elastomers generally contain at least 85 weight percent polyurethane segments and are generally called elasthane or spandex.

Methods of producing polyurethane elastomer fibers by various spinning operations are well known. Frequently polyurethane elastomer fibers are produced by a dry spinning process, for example fibers made of polyesterurethane/semicarbazide or polyesterurethane/diamine compositions, sold under the trademark Dorlastane; fibers made of polyetherurethane/hydrazine, polyetherurethane/diamine or polyesterurethane/diamine compositions, sold under the trademark Lycra; fibers made of polycaprolactone/diamine compositions sold under the trademark Asahi-Spandex; fibers made of polyetherurethane/diamine compositions, sold under the trademarks Asahi-Spandex or Espa; fibers made of polyetherurethane/aminohydrazide derivative compositions, sold under the trademark Neolon; or fibers made of polyetherurethane/hydrazine or polyetherurethane/diamine compositions, sold under the trademark Opelon. Polyurethane elastomer fibers can also be produced by a known wet spinning process. For example fibers made of polyester/polyetherurethane/diamine compositions, sold under the trademarks Numa or Fujiibo-Spandex are produced according to a wet spinning process. Furthermore, chemical spinning processes are also known and utilized, for example for the production of fibers made of polyesterurethane/diamine compositions, sold under the trademarks Glospan, Enkaswing or Spancelle.

Methods of producing polyurethane elastomers, for example via continuous polymerization, as well as useful starting materials for polyurethane elastomers are also well known in the art. Polyurethane elastomers, starting materials, synthesis of the elastomers, spinning operations etc. are described in Ullmanns Encyklopädie der technischen Chemie, 4th edition, vol. 11, pages 312-320.

According to the methods of the present invention

a) the lubricant is contacted with a polymeric starting material for the elastic fibrous material before or during the polymeric material is spun to the elastic fibrous material and/or  
 b) the lubricant is contacted with the produced elastic fibrous material.

Accordingly, the lubricant can be added prior to, during or after the spinning of filaments, fibers etc. from

a polymeric starting material, such as a polyurethane elastomer.

Methods of contacting a lubricant with a polymeric starting material before or during the polymeric material is spun to an elastic fibrous material are well known in the art. The abstracts of published Japanese patent applications J03287816 and J01282387 suggest kneading an oily agent, such as a siloxane or a mineral oil, with a polyurethane elastomer or a with a polyisocyanate prepolymer prior to a melt spinning step for producing elastic polyurethane yarn.

In the method of the present invention the lubricant can be mixed with a polymeric starting material for an elastic fibrous material, such as a polyurethane elastomer or a polyisocyanate prepolymer, prior to spinning according to the methods generally suggested in the published Japanese patent applications J03287816 and J01282387. The lubricant can be mixed with the polymeric starting material while it is still solid, already molten or dissolved in a solvent. Alternatively, the lubricant can be added to the spin bath when the fibrous material is spun according to a wet-spinning process. In this case the lubricant is contacted with the polymeric starting material in the spin bath.

Alternatively, the lubricant can be contacted with the produced elastic fibrous material, for example before or during drying of the spun material or before or during the elastic fibrous material is subjected to a twining process. The application of the lubricant prevents that the spun filaments, fibers, rovings etc. stick together and further prevents incrustation of the drying or twining device. The lubricant can be contacted with the elastic fibrous material in a generally known manner, for example by spraying the lubricant on the fibrous material or dipping the fibrous material into the lubricant.

Alternatively, or in addition to the method described above, the lubricant can be contacted with a fibrous material before or during the elastic fibrous material is subjected to a further processing step, such as a weaving or knitting process. The lubricant can be contacted with the fibrous material in a generally known manner, for example by spraying the lubricant on the fibrous material or dipping the fibrous material into the lubricant.

In any event, the polymeric starting material and/or the fibrous material is contacted with an effective amount of the lubricant. The amount which is effective for lubrication depends on various factors, such as the manner of applying the lubricant to the polymeric starting material or to the fibrous material, the specific components of the lubricant, the type of the polymeric starting material or the fibrous material, etc. Generally, from 0.1 to 20 percent, preferably from 1 to 10 percent, more preferably from 2 to 8 percent of an above-described polyether polyol is applied, based on the weight of the polymeric starting material or the fibrous material.

The polyether polyol described above which is comprised in the lubricant, is biodegradable and is generally miscible with water at any weight ratio. The water-miscibility or -solubility facilitates the removal of the lubricant from the elastic fibrous material when it is not desired on the elastic fibrous material any longer, for example after a cloth has been produced from the filaments, rovings, threads, yarns, strands etc. Yellowing of the fibrous material or articles like cloths produced therefrom is not observed.

The lubricant is further illustrated by the following examples which should not be construed to limit the scope of the invention. Unless otherwise mentioned, all parts and percentages are by weight.

#### Examples 1 to 8 and Comparative Examples A to G

40 The lubricant of Example 1 is a polyether polyol which is the reaction product of sorbitol and propylene oxide in a molar ratio of 1 : 9. In the closed bottle biodegradability test according to OECD-Nr. 301-D, 40% of the polyether polyol is degraded in 28 days.

The lubricant of Example 2 is a polyether polyol which is the reaction product of sorbitol and ethylene oxide at a molar ratio of 1 : 9.

45 The lubricant of Example 3 is a polyether polyol which is the reaction product of sorbitol with ethylene oxide at a molar ratio of 1 : 15.

The lubricant of Example 4 is a polyether polyol which is the reaction product of sorbitol with 1,2-butylen oxide and then with propylene oxide at a molar ratio of sorbitol : 1,2-butylen oxide : ethylene oxide of 1 : 4 : 4.

50 The lubricant of Example 5 is a polyether polyol which is the reaction product of sorbitol with 1,2-butylen oxide and then with propylene oxide at a molar ratio of sorbitol : 1,2-butylen oxide : ethylene oxide of 1 : 6 : 6.

The lubricant of Example 6 is a polyether polyol which is the reaction product of sorbitol with 1,2-butylen oxide and then with propylene oxide at a molar ratio of sorbitol : 1,2-butylen oxide : ethylene oxide of 1 : 6 : 8.

55 The lubricant of Example 7 is a reaction product of a sucrose/glycerine mixture and propylene oxide at a molar ratio of sucrose : glycerine : propylene oxide of 0.67 : 0.33 : 7.5.

The lubricant of Example 8 is a reaction product of a sucrose/glycerine mixture and propylene oxide at a

molar ratio of sucrose : glycerine : propylene oxide of 0.9 : 0.1 : 15.

The lubricant of Comparative Example A is a reaction product of butanol as initiator and an ethylene oxide/propylene oxide mixture at a molar ratio of ethylene oxide : propylene oxide of 50 : 5. The viscosity of the lubricant is 300 cSt at 50 °C. Its weight average molecular weight is about 3,000.

5 The lubricant of Comparative Example B is a reaction product of butanol as initiator and propylene oxide. The weight average molecular weight of the lubricant is about 700.

The lubricant of Comparative Example C is a reaction product of a C<sub>8</sub>-fatty alcohol as initiator with ethylene oxide and then propylene oxide at molar ratio of alcohol : ethylene oxide : propylene oxide of 1 : 8 : 2.

10 The lubricant of Comparative Example D is a reaction product of ethylene diamine as initiator and propylene oxide. The weight average molecular weight of the lubricant is about 350.

The lubricant of Comparative Example E is a reaction product of a mixture of C<sub>12-15</sub>-alcohols with propylene oxide and then ethylene oxide at molar ratio of alcohol : propylene oxide : ethylene oxide of 1 : 4 : 2.

The lubricants of Comparative Examples F and G are mineral oil and silicone SI-200 respectively.

15 The lubricating properties of the compounds of Examples 1 to 8 and the effect of these compounds on Lycra polyurethane fibers are listed in Table 1 below.

The lubricating properties are determined according to DIN 51 350, part 3 wherein the wearing characteristics of fluid lubricants in a SHELL four-ball tester are evaluated. The tester consists of one rotating ball which is subject to a load of 150 N and which slides on three stationary balls identical to it. The test period is one hour. The ball Imprint diameters of the three stationary is measured in mm. The lower the ball Imprint diameter is, the better is the lubricating property of the fluid lubricant.

20 The lubricants of Examples 1 to 8 and of Comparative Examples A to G are evaluated regarding their tendency to reduce the elasticity of polyurethane elastomers in the following manner: a cloth made of a yarn of about 0.1 mm thickness and produced from a mixture of 20% LYCRA polyurethane fibers and 80% cotton wool is stretched for 80%. Three drops of the lubricant to be tested are applied to the cloth and left there for 40 min. 25 at room temperature. The cloth is then placed in an oven at 180°C. The cloth is released and visually inspected regarding loss of elasticity.

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Table 1

5	Example	Wear/scar (150N)	Loss of stretch
10	1	0.25 mm	no 2)
15	2	0.24 mm	no 2)
20	3	0.26 mm	no 2)
25	4	0.26 mm	no 2)
30	5	0.24 mm	no 2)
35	6	0.24 mm	no 2)
40	7	0.3 mm	no 2)
45	8	0.25 mm	no 2)
50	A	0.60 mm	yes
55	B	0.55 mm	yes
60	C	0.47 mm	yes
65	D	0.5 mm	yes
70	E	0.47 mm	no 3)
75	mineral oil	...1)	no 3)
80	silicone Si-200	... 1)	no 3)

1) not measured

2) no yellowing of the cloth observed

3) yellowing of the cloth observed after removal of mineral oil or silicone Si-200

The polyether polyol of Example 1 is mixed with water or dipropylene glycol dimethyl ether at various weight ratios and the lubricating property of the mixtures is measured as described above and listed in Table 2 below.

Table 2

45	Polyether polyol of Example 1 (%)	Dipropylene glycol dimethyl ether (%)	Water (%)	Viscosity (cST at 40°C)	Wear/Scar (150N)
50	100	--	--	3755	0.25 mm
55	90	--	10	655	0.24 mm
60	85	--	15	165	0.26 mm
65	90	10	--	680	0.24 mm

Table 2 illustrates that the polyether polyols in the methods of the present invention have excellent lubricating properties, even when mixed with water or water-miscible diluents.

Example 9

5 35 parts of monopropylene glycol, 30 parts of tripropylene glycol methyl ether and 35 parts of a polyether polyol which is the reaction product of sorbitol and ethylene oxide in a molar ratio of 1 : 18 are mixed. The viscosity of the mixture is 22 cSt at 40 °C.

Example 10

10 35 parts of monopropylene glycol, 35 parts of tripropylene glycol methyl ether and 30 parts of the polyether polyol of Example 1 are mixed. The viscosity of the mixture is 22 cSt at 40 °C.

The mixtures of Examples 9 and 10 are evaluated regarding their tendency to reduce the elasticity of polyurethane elastomers as described above for Examples 1 - 8 and Comparative Examples A to G. Very little loss of stretch is observed. The loss of stretch is much lower than the loss of stretch observed for the lubricants of Comparative Examples A to E.

15 The lubricating properties of the mixtures of Examples 9 and 10 and their wear resistance at high temperatures are evaluated on a SRV (Schwingreibungsvorschleiss) apparatus which is available from the OPTIMOL company (Germany). A metal ball vibrates on a metallic disc. The metal ball is subject to a load of 200 N. After an hour both mixtures are still liquid which illustrates their excellent wear resistance.

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**Claims**

1. A method of producing a lubricated elastic fibrous material which method comprises contacting the fibrous material, or a polymeric starting material for the fibrous material, with a lubricant comprising a polyether polyol which is the reaction product of
  - i) one or more C<sub>2-4</sub>-alkylene oxides; and
  - ii) a polyhydroxy compound which is a pentite, a hexite, a monosaccharide, or a disaccharide.
2. The method of Claim 1, wherein the polymeric starting material for the fibrous material comprises a polyurethane elastomer.
3. The method of Claim 1 or Claim 2, wherein the polymeric starting material is contacted with the lubricant before or whilst the elastic fibrous material is subjected to a twining process.
4. The method of Claim 1 or Claim 2, wherein the elastic fibrous material is contacted with the lubricant before or whilst the elastic fibrous material is subjected to a knitting or weaving process.
5. The method of any one of Claims 1 to 4, wherein the elastic fibrous material is at least partially made of a polyurethane elastomer material.
6. The method of any one of Claims 1 to 5, wherein the molar ratio between the alkylene oxide and the polyhydroxy compound is from 6:1 to 18:1.
7. The use as a lubricant for an elastic fibrous material of a polyether polyol which is the reaction product of
  - i) one or more C<sub>2-4</sub>-alkylene oxides; and
  - ii) a polyhydroxy compound which is a pentite, a hexite, a monosaccharide, or a disaccharide.
8. An elastic fibrous material incorporating a lubricant comprising a polyether polyol which is the reaction product of
  - i) one or more C<sub>2-4</sub>-alkylene oxides; and
  - ii) a polyhydroxy compound which is a pentite, a hexite, a monosaccharide, or a disaccharide.
9. The elastic fibrous material of Claim 8, being at least partially made of a polyurethane elastomer material.
- 55 10. The elastic fibrous material of Claim 8 or Claim 9, wherein the molar ratio between the alkylene oxide and the polyhydroxy compound is from 6:1 to 18:1.



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## EUROPEAN SEARCH REPORT

Application Number  
EP 94 30 0431

DOCUMENTS CONSIDERED TO BE RELEVANT			CLASSIFICATION OF THE APPLICATION (Int.CLS)
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	
D, Y	FR-A-1 501 916 (ETABLISSEMENTS KUHLMANN) * page 3, left column, paragraph 5; claims * ---	1-10	D06M13/17 D06M15/03 D06M13/165 D06M15/53
D, Y	US-A-3 370 056 (MASARU YOTSUZUKA ET AL) * column 1, line 24 - line 33 * * column 3, line 15 - line 43; claims 1,2 * ---	1-10	
D, Y	US-A-4 496 632 (CAMP ET AL) * column 1, line 53 - column 2, line 9; claim 16 * ---	1-10	
D, Y	WPIL, FILE SUPPLIER, DERWENT PUBLICATIONS LTD.; LONDON, GB.; AN=89-375208 & JP-A-1 282 387 (KANEBO KK) 14-11-1989 * abstract *---	1-10	
D, Y	US-A-3 704 225 (BARRY MICHAEL SHAY) * abstract; claims *---	1-10	TECHNICAL FIELDS SEARCHED (Int.Cl.5)
D, A	US-A-3 421 935 (NEIL L. FINCH) * the whole document *-----	1-10	C10M D06M C07H
The present search report has been drawn up for all claims			
Place of search	Date of completion of the search	Examiner	
THE HAGUE	24 May 1994	Blas, V	
CATEGORY OF CITED DOCUMENTS		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons A : member of the same patent family, corresponding documents	
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document			